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# THE USE OF PHENOTHIAZINE AS AN INSECTICIDE

By L. E. Smith, Division of Insecticide Investigations

## INTRODUCTION

Phenothiazine, first prepared in 1885 by Bernthsen, is the parent substance of a series of important dyes, among which is the very valuable methylene blue. The insecticidal properties of this compound were first reported by workers in the Bureau of Entomology and Plant Quarantine during the course of an investigation of synthetic organic compounds, especially those containing nitrogen and sulphur, as possible substitutes for the arsenicals.

Although phenothiazine, in common with other organic insecticides, is specific in its action on insects (highly toxic to some, practically nontoxic to others), preliminary results of tests with it against some of our most destructive insect pests have been so favorable as to make it worthy of testing against other insects. This review has been prepared to assist entomologists seeking new means of combating insects. It is hoped that it will stimulate further work with a very promising insecticide.

The use of phenothiazine as an insecticide is still in the experimental stage. The results of tests against various insects which are recorded in this review are only preliminary, and no recommendations with regard to the use of this compound as an insecticide can be made at this time. A great deal of unpublished work (reports from Bureau entomologists) is recorded in this review. These unpublished reports are not available for distribution, but when more extensive insecticidal studies have been made the results will probably be published by personnel engaged in this work.

## ORIGINAL TESTS

Phenothiazine was first tested as a possible insecticide against mosquito larvae by Bulger (6, 8), who found that it was more toxic than rotenone, being effective at a concentration of 1:1,000,000. These results were so promising that the Division of Control Investigations made tests on a variety of insects, with the following results: Phenothiazine in benzene and acetone solutions was not toxic to house flies. Against silkworms a dose of 0.06 mg per gram of body weight by the sandwich method gave a rapid kill, indicating that phenothiazine is considerably more toxic than lead arsenate. In toxicity to the tent caterpillar phenothiazine was superior to lead arsenate. Phenothiazine was not effective against mealybugs on coleus and Jerusalem cherry.

Foliage tests on plum, apple, peach, quince, coleus, beans, Jerusalem cherry, and citrus seedlings showed no material injury by sprays containing 2 pounds of phenothiazine in 50 gallons of water.

Tests on house flies in another laboratory (12) verified the fact that phenothiazine was not toxic when applied in benzene or acetone solution, but showed that when applied as a dust it was very toxic in 24 hours and had no repellent action. Tests of other workers (11) also showed phenothiazine to be very toxic to the silkworm.

The use of synthetic organic compounds as insecticides to replace the arsenicals is discussed in the Annual Report of the Chief of the Bureau of Entomology and Plant Quarantine, 1935 (18).

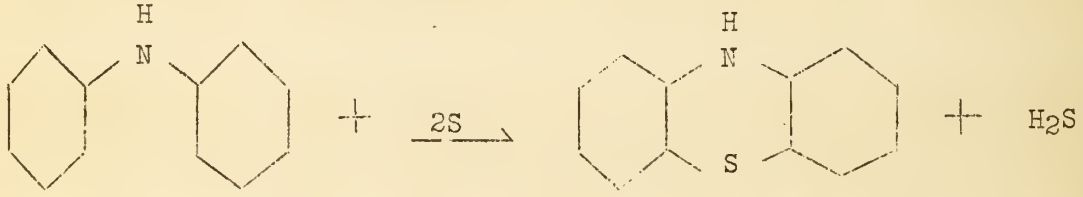
The possibilities of phenothiazine as an insecticide have been mentioned in the Bureau of Entomology and Plant Quarantine News Letter at various times. A brief summary of its toxicity to codling moth and other lepidopterous larvae is found in the May 1934 issue.

Brief mention has been made of phenothiazine, or of the type of synthetic organic compounds of which it is a member, as a possible insecticide (14, 15, 16).

Popular articles have appeared pointing out the possible uses of phenothiazine as an insecticide (1, 2, 3, 9, 17).

## CHEMISTRY OF PHENOTHIAZINE

Phenothiazine is a product of the reaction between diphenylamine and sulphur, which can be expressed as follows:




To improve the yield of phenothiazine and to lower the temperature at which the reaction takes place, various catalysts have been employed. For production in quantity it has been found that when a trace of iodine is added the reaction proceeds almost quantitatively at about 180° C. The resulting material is practically 100 percent pure. When recrystallized from a suitable solvent, such as benzene or toluene, it occurs as light yellow leaflets melting at 180° C.

## TOXICITY TO MAMMALS

Acute toxicity of phenothiazine to warm-blooded animals is so low that fatal doses have not been determined; therefore, the margin of safety appears to be ample. When administered orally or gastrically to rats, rabbits, and man, phenothiazine acts as a urinary antiseptic, as shown by effects upon experimental cystitis in rabbits and by preliminary clinical trials (4, 5, 10).

## TOXICITY TO BEES

Somerset, Md., Bee Culture Laboratory (Division of Fruit Insect Investigations. Results of codling moth investigations, 1934, Part II, pp. 33-34. Mimeo.).--Phenothiazine as a stomach poison had a slower action on honeybees than lead arsenate, but phenothiazine was toxic to bees in field experiments.

## INSECTICIDAL TESTS AGAINST VARIOUS INSECTS

### Mexican Bean Beetle

Ohio (N. F. Howard, L. W. Brannon, and H. C. Mason. Derris and other insecticides for the control of the Mexican bean beetle. Jour. Econ. Ent. 28: 444-448. 1935).--Phenothiazine at the rate of 3 pounds to 50 gallons of water gave good control of the Mexican bean beetle in two experiments during 1934. In one experiment in which the material was used at the rate of 1 pound to 50 gallons the control was only fair. No noticeable injury to bean foliage resulted.

Olney, Md. (L. W. Brannon. Annual report, Norfolk, Va., laboratory, Division of Truck Crop and Garden Insect Investigations. 1935. Typewritten).--Phenothiazine gave good protection when used as a spray, 2 pounds to 50 gallons, but the control was poor when used as a dust at the rate of 1 part to 10 parts of talc.

South Point, Ohio (N. F. Howard and H. C. Mason. Annual report, Columbus, Ohio, laboratory, Division of Truck Crop and Garden Insect Investigations, pp. 2-75. 1935. Typewritten).--In many field experiments phenothiazine proved very effective as a spray at a concentration of 2 pounds to 50 gallons. The control ranked with that obtained with derris and cube. At a concentration of 1 pound to 50 gallons the control was good. When used as a 1:10 dust, however, the control was poor. Slight foliage injury was observed shortly after application, but the injury appeared to be only temporary.

Columbus, Ohio (N. F. Howard and H. C. Mason. Annual report, Columbus laboratory, Division of Truck Crop and Garden Insect Investigations, p. 87. 1935. Typewritten).--When used as a spray at a concentration of 2 pounds to 50 gallons, the control was very satisfactory. When used at 1 pound to 50 gallons, the control was only fair when the infestation of the bean beetle was most severe. As a dust, diluted 1 to 5 with talc, the control was fair in the only experiment carried out. However, a 1:10 dust proved to be ineffective. A temporary foliage injury was observed.

Urbana, Ill. (W. P. Flint, Illinois Natural History Survey and Agricultural Experiment Station, letter to L. A. Strong, Chief, Bureau of Entomology and Plant Quarantine, May 8, 1936).--Phenothiazine used as a dust gave fair to very good results in tests for the control of the Mexican bean beetle in 1935.

## Tobacco Hornworm

Quincy, Fla. (F. S. Chamberlain. Annual report, Quincy laboratory, Division of Truck Crop and Garden Insect Investigations, pp. 9, 24, 28-32. 1935. Typewritten).--Preliminary tests with phenothiazine diluted 1:10 with filter dust showed no foliage injury to tobacco plants. However, in field tests this dust failed to control the hornworm. In cage tests at concentrations from 0.062 to 2 percent phenothiazine was much less toxic than lead arsenate. Both field and cage experiments indicate that phenothiazine shows little promise as a satisfactory controlling agent of the hornworm.

Clarksville, Tenn. (J. U. Gilmore. Annual report, Clarksville laboratory, Division of Truck Crop and Garden Insect Investigations. 1935. Typewritten).--In cage experiments the results using phenothiazine as a dust were not promising.

## Cotton Insects

Tallulah, La. 1934. (M. T. Young and G. L. Smith. Field-plot and cage tests for boll-weevil control. Jour. Econ. Ent. 29: 105-111. 1936).--Field tests of phenothiazine 1:9 with dusting sulphur had little, if any, effect on the boll-weevil infestation, but a plot treated with the mixture yielded 60 pounds of seed cotton per acre more than the untreated plot. This amount, however, is not considered significant. In the cage tests the percentage mortality with 1 part of phenothiazine to 9 parts of sulphur was greater than in the untreated cages but much less than when calcium arsenate was used.

1935. (G. L. Smith, J. C. Clark, and A. L. Scales. Comparative tests of phenothiazine mixtures with sulphur and clay, calcium arsenate, and check for cotton boll weevil and leafworm control. Annual report, Tallulah laboratory, Division of Cotton Insect Investigations. 1935. Typewritten. See also Bur. Ent. and Plant Quar. News Letter October 1935, p. 17, and May 1936, pp. 13 and 16).--In cage tests phenothiazine alone and in various mixtures with sulphur and clay was not so effective as calcium arsenate when used against the cotton boll weevil. When used against the cotton leafworm phenothiazine and various mixtures with sulphur and clay proved to be ineffective. Phenothiazine mixed 1:9 with sulphur when used for control of the boll weevil gave an increase in yield of cotton of 17.5 percent as compared with a 23.7 percent increase when calcium arsenate was used.

Tucson, Ariz. (T. P. Cassidy and T. C. Barber. Hemipterous insect investigations in Arizona crop season. Annual report, Tucson laboratory, Division of Cotton Insect Investigations, 1935. Typewritten).--When used against five species of hemipterous insects on cotton, phenothiazine was not effective.

Port Lavaca, Tex. (K. P. Ewing and R. L. McGar. Quarterly report, Port Lavaca laboratory, Division of Cotton Insect Investigations, July-September, 1935. Typewritten).--When used as a dust full strength or mixed with equal parts of clay, phenothiazine gave poor control of adults and nymphs of the cotton fleahopper.

## Lima Bean Pod Borer

Ventura, Calif. (R. Cecil. Annual report, Ventura laboratory, Division of Truck Crop and Garden Insect Investigations, 1935. Typewritten).--In five experiments using phenothiazine diluted 1:10 with diatomaceous earth as a dust, the average control was 71.15 percent. This figure is reported to be of little significance owing to light infestation and other conditions that are set forth in the report.

## Grape Berry Moth

Venice, Ohio (Division of Fruit Insect Investigations. Results of codling moth investigations, 1934, Part II, p. 31. Mimeo.).--Phenothiazine 4 pounds per 100 gallons with 4 pounds of bentonite and 1 quart of fish oil gave 7.0 percent of wormy grapes, while calcium arsenate 2.5 pounds per 100 gallons, bordeaux mixture 2-4-50, and 1 pint of fish oil gave 6.7 percent of wormy grapes.

Sandusky, Ohio (G. A. Runner. Annual report, Sandusky laboratory, Division of Fruit Insect Investigations, 1935. Typewritten).--In one series of field experiments phenothiazine was used at a strength of 4 pounds in 100 gallons of water in three applications. In the first two applications fish oil was added at a strength of 1 pint per 100 gallons. The percentage of damaged berries was 18.5, as compared with 19.9 when calcium arsenate was used. Because of the possibility that the injury to grapes might have been caused by the combination of phenothiazine with fish oil, in mid-August special experiments were started with phenothiazine alone compared with fish oil. No injury of consequence appeared in either of these tests.

## Codling Moth -- Laboratory Tests

Takoma Park, Md. (L. E. Smith, F. Munger, and E. H. Siegler. Phenothiazine. A promising new insecticide. Jour. Econ. Ent. 28:727-728. 1935).--The toxicity of phenothiazine to mosquito larvae was so high that members of the Division of Fruit Insect Investigations made laboratory tests with this material against codling moth larvae. Preliminary tests indicated that phenothiazine was a potential insecticide for codling moth control, being as effective as lead arsenate when used at the same concentration.

(E. H. Siegler, F. Munger, and L. E. Smith. Laboratory tests of phenothiazine against codling moth larvae. Jour. Econ. Ent. 28:532-536).--A large number of tests were then made using phenothiazine in combination with oil, bentonite, various fungicides, etc. The results indicate that phenothiazine was not compatible with oil, bentonite, and bordeaux mixture.

Vincennes, Ind. (Division of Fruit Insect Investigations. Results of codling moth investigations, 1934, Part II, p. 21. Mimeo.).--About 35 tests were made with phenothiazine in 1934. At 1/2 pound to 50 gallons it had an average efficiency of 63.8 percent, at 1 pound to 50 gallons 85.8 percent, and at 2 pounds to 50 gallons 91.5 percent. Flotation sulphur and soap lowered the efficiency slightly. A 2-4-50 copper phosphate-lime mixture had no effect on the toxicity of phenothiazine. A marked loss in toxicity occurred when bordeaux mixture or bentonite was added. There were very few stings with any phenothiazine treatment.

Yakima, Wash. (E. J. Newcomer. Annual report, Yakima laboratory, Division of Fruit Insect Investigations, 1935. Typewritten).--Phenothiazine was found to be ineffective as an ovicide, but gave good control when used as a larvicide.

Codling Moth--Field Tests 1934

Kearneysville, W. Va. (Division of Fruit Insect Investigations. Results of codling moth investigations, 1934, Part II, pp. 4-5. 1934. Mimeo.).--Phenothiazine was used at a concentration of 4 pounds per 100 gallons with 4 pounds of bentonite and 1 quart of fish oil. Some injury was noticed, and the fruits carried a heavy black residue which could not be removed by commercial methods.

Parma, Idaho (Division of Fruit Insect Investigations. Results of codling moth investigations, 1934, Part II, p. 13. 1934. Mimeo.).--A limited test was conducted with phenothiazine. The single count tree used showed 284 worms per 100 apples as compared with 241 worms when lead arsenate was used. The fruits were very spotted when the residue was rubbed off.

Codling Moth--Field Tests 1935

The phenothiazine used in all field tests in 1934 was subsequently proved by chemical and biological examination (7, 18) to be only 55 percent pure, and the 45 percent of impurities had no toxic properties. In 1935 the method of preparation of phenothiazine was modified so that it was possible to procure a chemically pure material, which was used exclusively in the tests of that year. The material contained 2 percent of sulphated lauryl alcohol as a wetting agent. It is believed that this amount of wetter caused excessive run-off when used in the field.

Yakima, Wash. (Division of Fruit Insect Investigations. Results of codling moth investigations, Part II, pp. 2-4. 1935. Mimeo. See also Bur. Ent. and Plant Quar. News Letter, October 1935, pp. 4-5. Mimeo.).--When used at a concentration of 4 pounds per 100 gallons, phenothiazine gave 4.5 percent wormy fruit and 1.7 percent stings, or 94 percent clean fruit, as compared with 10.5 percent wormy fruit and 35.4 percent stings, or 59.3 percent clean fruit, when lead arsenate was used at a concentration of 3 pounds per 100 gallons. The fruits were smaller and the ground color was a darker green when phenothiazine was used.

Vincennes, Ind. (Division of Fruit Insect Investigations. Results of codling moth investigations, Part II, pp. 12-14. 1935. Mimeo.).--Phenothiazine was one of the most toxic materials ever tested at the Vincennes laboratory, although in the form used it appeared to be susceptible to weathering and possibly some decomposition. With a single application phenothiazine showed a higher initial toxicity than lead arsenate. Phenothiazine caused severe injury when used with oil, and serious residue difficulties and some loss in toxicity when used with bentonite or bordeaux. In experiments with second-brood larvae the phenothiazine-soap combination was equal to lead arsenate-bordeaux. There was a significant decrease in the number of stings when phenothiazine was used. During hot weather workers were burned on face and arms.

St. Joseph, Mo. (Division of Fruit Insect Investigations. Results of codling moth investigations, Part II, pp. 19-20. 1935. Mimeo.).--Phenothiazine at a concentration of 3 pounds per 100 gallons gave 85.1 percent clean fruit (13.7 percent wormy; 3.9 percent stings), and lead arsenate at the same dosage gave 84.5 percent clean fruit (4.8 percent wormy and 14.8 percent stings). There was no visible injury to foliage, but there was a retardation of the normal coloring of the fruit. Phenothiazine caused some burning of the face and arms of workers in hot weather.

Kearneysville, W. Va. (Division of Fruit Insect Investigations. Results of codling moth investigations. Part II, pp. 25-26. 1935. Mimeo.).--The percentage of sound fruit was about the same for phenothiazine-bordeaux and the standard lead arsenate treatment, but the control was less satisfactory owing to the much higher proportion of wormy fruit when phenothiazine was used, which would result in a heavier carry-over of worms. The phenothiazine residue was very heavy and dark green in color, but no injury was noted.

Newark, Del., State Experiment Station. (Division of Fruit Insect Investigations. Results of codling moth investigations, Part I, p. 10. 1935. Mimeo.).--Phenothiazine was decidedly less effective than lead arsenate owing either to wash-off by heavy rains, as it was used without sticker, or to deterioration.

Wooster, Ohio, State Agricultural Experiment Station. (Division of Fruit Insect Investigations. Results of codling moth investigations, Part I, p. 42. 1935. Mimeo.).--Phenothiazine was used in field tests and, while owing to low infestation the results were inconclusive, the material appears to be worthy of further trials. No injury was noted on fruits or foliage, but the addition of a fungicide would be necessary for its use in Ohio.

Urbana, Ill. (W. P. Flint, Illinois Natural History Survey and Agricultural Experiment Station, letter to L. A. Strong, Chief, Bureau of Entomology and Plant Quarantine, May 8, 1936).--In orchard experiments phenothiazine was found to be incompatible with oil. Used alone it gave very good results during the season of 1935, but the results at the end of the season were poor owing to lack of late applications.

A brief summary of the results obtained by the Bureau of Entomology and Plant Quarantine with phenothiazine when used against the codling moth has appeared (13).

## Apple Maggot

New Haven, Conn. (P. Garman. Studies in breeding and control of the apple maggot. Conn. Agr. Expt. Sta., Rept. of Entomologist. 1935: 315-320.).--In laboratory tests phenothiazine had considerable repellent action towards oviposition by the apple maggot.

## Plum Curculio

Fort Valley, Ga. (O. I. Snapp. Annual report, Fort Valley laboratory, Division of Fruit Insect Investigations. 1935. Typewritten).--Laboratory tests with phenothiazine at strengths of 1, 2, and 4 pounds per 100 gallons showed no toxicity to the plum curculio.

Tomato Fruit Worm

Quincy, Fla. (F. S. Chamberlain. Annual report, Quincy laboratory, Division of Truck Crop and Garden Insect Investigations, pp. 9, 24, 28-32. Typewritten).--In field experiments phenothiazine 1:10 with filter dust gave only slight control of the tomato fruit worm.

Cabbage Worm

Columbus, Ohio (N. F. Howard and H. C. Mason. Annual report, Columbus laboratory, Division of Truck Crop and Garden Insect Investigations, pp. 108-125. 1935. Typewritten).--When used as a spray at 1 and 2 pounds per 100 gallons and as a dust at 1 part to 10 parts of talc, phenothiazine was ineffective against cabbage worms. There was a slight injury to the foliage, which had a spotted appearance.

Japanese Beetle

Moorestown, N. J. 1934 (W. E. Fleming and F. E. Baker. Report on derris and its constituents as insecticides and repellents against the Japanese beetle along with tests with miscellaneous materials. Division of Japanese Beetle Investigations. 1934. Typewritten).--A suspension of 2 to 4 pounds of phenothiazine per 100 gallons of water with wheat flour had a slight narcotic effect on the Japanese beetle when sprayed on foliage. When used at the rate of 32 pounds to 100 gallons, the beetles coming in contact with or eating the material passed into a stupor which lasted several hours. Upon reviving, however, the beetles returned to the foliage. When phenothiazine was added to derris, the effectiveness of derris as a repellent appeared to decrease.

1935 (W. E. Fleming and F. E. Baker. Report on homologs of paris green, compounds of the alkali and alkaline earth metals, derris, rotenone, organic sulphur compounds, and miscellaneous materials as insecticides agains the Japanese beetle, Division of Fruit Insect Investigations. 1935. Typewritten).--Crude phenothiazine at the rates of 2, 4, 8, 16, and 32 pounds to 100 gallons and the pure material at the rates of 8, 16, and 32 pounds to 100 gallons were not effective as a stomach poison against the Japanese beetle and had only a slight repellent action. The feeding was slight, however, when pure phenothiazine was used at a rate of 32 pounds per 100 gallons.

European Corn Borer

New Haven, Conn. (C. H. Batchelder. Infestation of dahlias by the European corn borer and investigations of the insecticidal method of protection. Report, New Haven laboratory, Division of Cereal and Forage Insect Investigations. 1935. Typewritten).--When used as a spray on dahlia plots infested with the corn borer, pheonthiazine at a concentration of 4 pounds to 100 gallons reduced the infestation 92.4 percent, being as effective as when used earlier in the season against the ear worm on sweet corn.

(N. Turner. Insecticides to control the European corn borer. Conn. Agr. Expt. Sta., Circ. 114, p. 75. 1936).--Phenothiazine when used in suspension at the rate of 2 pounds in 50 gallons of water with a suitable spreader was very effective against the European corn borer.

Glastonbury, Conn. (C. H. Batchelder and D. D. Questel. Annual report, European corn borer research, Division of Cereal and Forage Insect Investigations, pp. 167, 172, 175. 1935. Typewritten).--Phenothiazine was tested in both laboratory and field experiments in 1935. Effective protection was provided when the material was applied as a water suspension 2 pounds to 50 gallons, but the control was poor when the material was applied as a dust 1:32 with talc. As a spray it reduced the borer population 90 percent as compared with 40 to 44 percent reduction in the dust plots. The use of phenothiazine as a spray has taken a place with fixed nicotine and ground derris root as a European corn borer insecticide.

## Corn Ear Worm

New Haven, Conn. (G. W. Barber. Special report on corn ear worm, insecticide studies. Division of Cereal and Forage Insect Investigations. 1935. Typewritten).--When used as a spray at concentrations up to 4 pounds to 50 gallons with various common spreading agents, phenothiazine was ineffective. At a concentration of 4 pounds to 50 gallons and used with such stickers as powdered whole milk, gum tragacanth, molasses, bentonite, sulphonated castor oil, fish oil, and casein cement, phenothiazine gave an 87 percent kill and was more effective than nicotine tannate or rotenone. When used as a spray in 12 tests, phenothiazine gave an average of 26.7 percent uninfested ears. When used as a dust the average percentage of uninfested ears was 96.8, which was the control obtained when lead arsenate was used. The concentration of phenothiazine dust ranged from pure material to dilutions 1:8 with talc, with no loss in effectiveness.

## Tomato Pinworm

Pennsylvania (C. A. Thomas. Status of the tomato pinworm Gnorimoschema lycopersicella Busck in Pennsylvania. Jour. Econ. Ent. 29: 313-317. 1936).--Phenothiazine, 1 pound to 50 gallons of water, was toxic to pinworms on tomato plants when the plants were dipped in the suspension.

## Fire Brat

Ames, Iowa (B. T. Snipes, R. E. Hutchins, and J. A. Adams. Effectiveness of sodium fluoride, arsenic trioxide and thiodiphenylamine as food poisons for the fire brat. Jour. Econ. Ent. 29:421-426. 1936).--Phenothiazine had little toxic effect on the fire brat when used at concentrations up to 20 percent of its preferred food. Only a few insects died after 1 week's feeding under controlled conditions.

## Screw Worms

Dallas, Tex., and Valdosta, Ga. (R. Melvin. Quarterly report, Division of Insects Affecting Man and Animals, June 30, 1936. Typewritten).--When used as a dust on Cochliomyia americana eggs on wounds of warm-blooded animals, phenothiazine did not prevent the eggs from hatching but did prevent the larvae from becoming established.

(E. W. Laake. Annual report, Dallas laboratory, Division of Insects Affecting Man and Animals, 1936. Typewritten. See also Bur. Ent. and Plant Quar. News Letter, May 1936).--Phenothiazine appears to be very promising as a larvicide against the screw worm, and a summation of preliminary experiments is of record.

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